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CONTROLLED GRAIN BOUNDARY STRUCTURES IN SUPERCONDUCTORS.(U)

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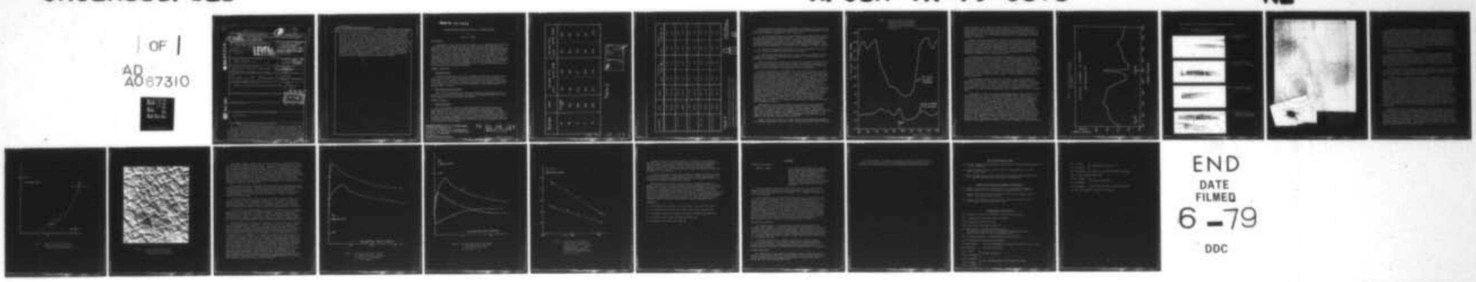

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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER AFOSR-TR-79-0373	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) CONTROLLED GRAIN BOUNDARY STRUCTURES IN SUPERCONDUCTORS.		5. TYPE OF REPORT & PERIOD COVERED Annual Technical Report, ~~1/1/78 to 12/31/78~~ 1 Jan - 31 Dec 78
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) E.J. Kramer		8. CONTRACT OR GRANT NUMBER(s) AFOSR-77-3107
9. PERFORMING ORGANIZATION NAME AND ADDRESS Cornell University Ithaca, New York 14853		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS 61102F 2306/C1
11. CONTROLLING OFFICE NAME AND ADDRESS AF Office of Scientific Research/NE Bldg. 410 Bolling AFB, Washington D.C. 20336		12. REPORT DATE February 1979
		13. NUMBER OF PAGES 21
14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office) Edward J. Kramer		15. SECURITY CLASS. (of this report) Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

"Approved for public release; distribution unlimited" 22p.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

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18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Superconductors

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The relationship between grain boundary structure and chemistry and the critical current properties of superconductors is being investigated. The critical current (flux pinning) contributed by single grain boundaries in Nb bicrystals has been observed and shown to be suprisingly large even for boundaries that should have no crystal anisotropy or stress field contribution to the elementary interaction force between a grain boundary and the flux line lattice. Transmission electron microscopy and X-ray topography have been used to charac-

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terize the grain boundary structure, and bicrystal substructure respectively. The first high angle symmetric tilt examined is straight, shows no periodic structure and appears to be free of precipitates or detectable segregation. The electron float zone welding method of bicrystal manufacture is shown to give rise to very low angle subboundaries lying approximately parallel to the main grain boundaries. These contribute an annoyingly high background critical current which makes isolation of the main boundary contribution somewhat uncertain. The effects of grain size and grain boundary segregation on flux pinning were investigated in Pb_{82} Bi_{18} polycrystalline films. It was demonstrated for the first time that segregation to the grain boundaries can markedly entrance the critical current density. Artificially segregated boundaries were produced by allowing Pb of Tl from an evaporated overlayer to diffuse in along the grain Boundaries. These results suggest new ways to process commercial superconductors for maximum flux pinning.

AFOSR-TR- 79-0373

CONTROLLED GRAIN BOUNDARY STRUCTURES IN SUPERCONDUCTORS

by

Edward J. Kramer

INTRODUCTION

Improvements in the critical current density J_c of commercial superconductors offer potential weight/volume savings for superconducting power machines (e.g., generators) that would make these particularly attractive for airborne applications. Pinning of flux lines in the superconductor by various crystal imperfections gives rise to a pinning force density $F_p = (\underset{\sim}{J}_c \times \underset{\sim}{B})$ and thus to the critical current density itself. There is strong circumstantial evidence that the important pinning imperfections in many commercial superconductors are grain boundaries, yet the fundamental mechanism of grain boundary flux pinning is in doubt. AFOSR-supported work on this project was begun on January 1, 1977 to investigate these fundamentals. This knowledge should allow new strategies to be devised for metallurgical optimization of flux pinning by grain boundaries (e.g., by controlling polycrystalline texture and/or grain boundary segregation).

RESEARCH OBJECTIVES

1. Bicrystal Studies

Produce, and characterize the grain boundary structure in, bicrystals which have different flux pinning contributions from different possible fundamental mechnaisms, i.e., the stress field interaction, the crystalline anisotropy interaction and the $\Delta\kappa$ interaction. Measure flux pinning by the boundary by measuring the variation of J_c as the angle between the magnetic field and the plane of the grain boundary is varied.

2. Thin Film Polycrystal Studies

Produce thin film polycrystals with grain boundaries predominantly normal to the film. Investigate the effects of grain size and impurity segregation to the grain boundaries on grain boundary flux pinning.

RESEARCH PROGRESS

1. Bicrystal Studies

An electron beam float zone welding technique for making macroscopic Nb bicrystals reported last year was further developed and bicrystals with high angle symmetric twist and tilt grain boundaries were produced (Table I). These bicrystals have no crystal anisotropy contribution to the elementary pinning force f_p [f_p is the force of interaction between a single grain boundary and the FLL]. In addition high angle asymmetric tilt boundary bicrystals were produced [Table 2] which have a substantial anisotropy contribution to f_p. These represent the first such bicrystals of Nb ever made.

Grant no. AFOSR-77-3107

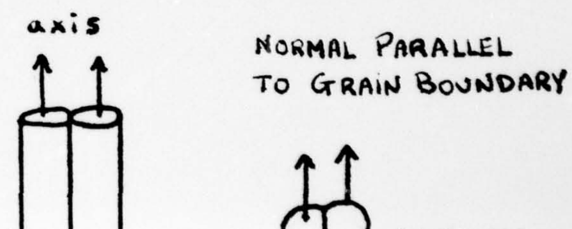
BICRYSTAL	TYPE	CRYSTAL # 1 axis	CRYSTAL # 1 normal ∥ G.B.	CRYSTAL # 2 axis	CRYSTAL # 2 normal ∥ G.B.
A	Symmetric tilt	211	231	211	231
B	tilt	211	231	211	231
C	tilt	211	111	211	111
1	tilt	211	231	211	111
2	tilt	211	110	211	110

Table 1

BICRYSTAL	TYPE	ANGLE	CRYSTAL #1 AXIS	CRYSTAL #1 NORMAL ‖ G.B.	CRYSTAL #2 AXIS	CRYSTAL #2 NORMAL ‖ G.B.
	TILT	19° 28'	110	211	110	111
	TWIST	19° 28'	111	211	211	111
B.C. # 9	TILT	30° 00'	110	211	110	110
	TWIST	30° 00'	110	211	211	110
B.C. #3 , #8	TILT	35° 16'	110	111	110	110
	TWIST	35° 16'	211	111	100	110
B.C. #6	TILT	45° 00'	110	100	110	110
	TWIST	45° 00'	110	100	221	110
B.C. #4,#5,#7	TILT	54° 44'	110	100	110	111
	TWIST	54° 44'	110	100	211	111

Table 2

List of bicrystals with a substantial crystal anisotropy contribution. Bicrystals 3-9 have already been produced while the others are



The bicrystals are being used in experiments to determine the relative sizes of the three feasible interaction mechanisms for the grain boundary-flux line lattice (FLL) interaction giving rise to f_p. Those are:

a) The interaction between the grain boundary stress fields and the strain field of the FLL (stress field interaction). Transmission electron microscopy on selected boundaries in Tables I and II reveals no resolvable dislocation structure (primary or secondary) to these high angle boundaries (although there are occasionally dislocations from the matrix that thread the boundary). Hence the stress field interaction is not expected to be important for these boundaries.

b) The grain boundary/FLL interaction due to the anisotropy of the upper critical field H_{c_2} (crystalline anisotropy interaction).

c) The grain boundary/FLL interaction due to electron scattering from the boundary which changes the Ginzburg-Landau parameter κ in the vicinity of the boundary ($\Delta\kappa$ interaction).

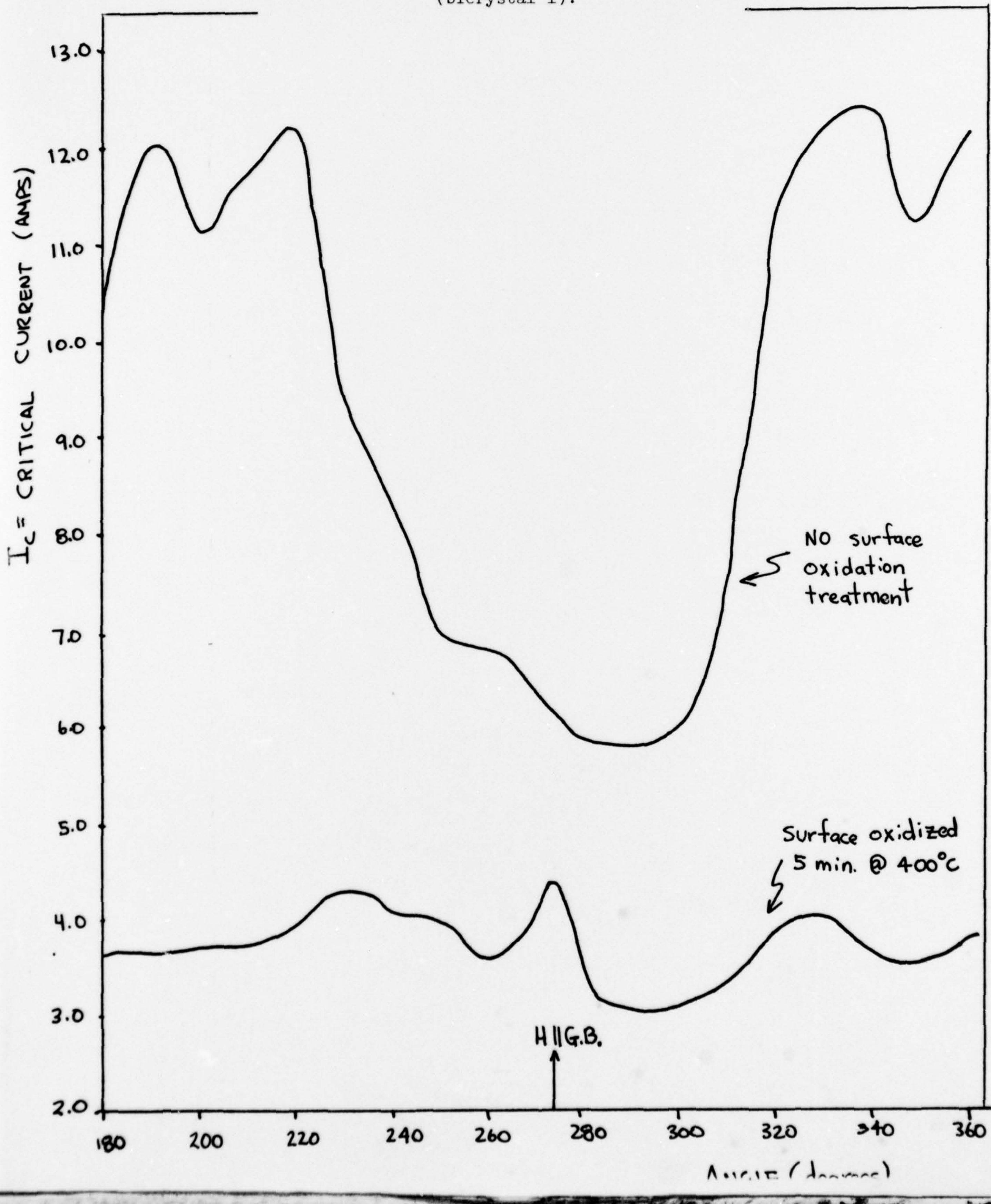
Since the bicrystals in Table I have only the $\Delta\kappa$ interaction where those in Table II have both $\Delta\kappa$ and crystalline anisotropy interactions, a comparison between the flux pinning due to the grain boundary in the two types of bicrystals should lead to an estimate of the importance of each interaction.

Several complications arise however due to the presence of lattice defects other than the bicrystal grain boundary. If one is not careful the pinning due to these will swamp the pinning due to the single boundary. One such unavoidable defect which is a strong flux pinning center is the specimen surface. It was found that oxidizing the crystal surface by heating it for 5 minutes to 400°C in air was very effective in removing surface pinning. Oxidizing for longer times did not further change the pinning (specifically it did not lead to further enhancement of the grain boundary pinning peak). The surface pinning could be recovered by chemically polishing off the surface layer and removed again by reoxidizing without altering the grain boundary pinning peak. These observations strongly indicate that the grain boundary peak observed is due to the intrinsic pinning of the boundary and not due to oxygen segregation to, or oxide penetration down, the grain boundary. [Unlike the substitutional impurities, interstitial impurities such as oxygen should not diffuse faster down the grain boundary than in the perfect lattice; in fact interstitial grain boundary diffusion may be slower due to trapping of interstitials in regions of different grain boundary structure.]

Measurements of the critical current as a function of angle between the magnetic field H and the plane of the grain boundary reveal a peak when the direction of H (and thus the FLL) is parallel to the boundary. The height of this peak is a measure of $\tilde{f}_p$ of the boundary where $\tilde{f}_p$ is the elementary interaction force per unit grain boundary area [Numerically $\tilde{f}_p = \Delta I_c\, B/\ell$ where ΔI_c is the height of the critical current peak, B is the magnetic induction and ℓ is the length of the boundary along the field direction.].

Figure 1 shows such a peak in a oxygen surface treated high angle symmetric tilt bicrystal measured at a field of .22T. The $\tilde{f}_p$ that can be inferred from this

Figure 1. Critical current versus angle between magnetic field direction and an arbitrary axis normal to a 46° tilt bicrystal (bicrystal 1).



peak is $\sim$80 N/m^2. This pinning is suprisingly strong, within a factor of 20 of the pinning expected from the external surface. Since this boundary has no anisotropy or stress field contribution, the pinning will be due entirely to the $\Delta\kappa$ interaction. All other high angle boundaries we have been able to measure at fields well below H_{c2} ($\simeq$.3T) also show a peak that can be interpreted as a grain boundary pinning peak.

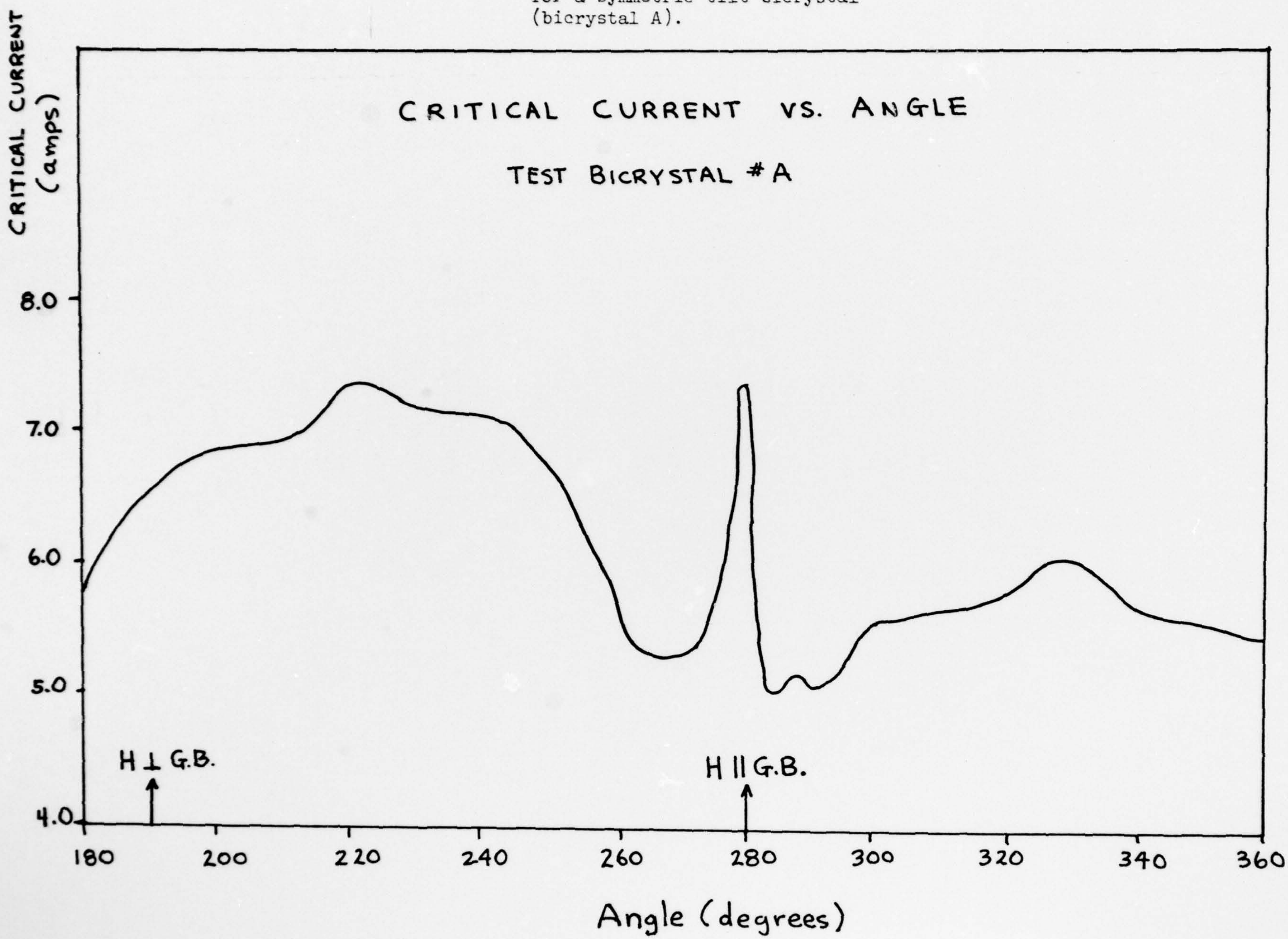
However, the pinning measurements also show an anisotropy that cannot be attributed to the grain boundary. (In Figure 1 there are other broader peaks. This non-uniform background is observed in the pinning anisotropy of all other bicrystals. In some cases the background makes it difficult to attribute peaks in pinning the grain boundary, although the true grain boundary peaks are usually rather more sharp than peaks in the background. Figure 2 shows an unusually sharp grain boundary peak. In any case the uneven background contributes considerable uncertainty to the actual peak height.

Consequently we have made an effort to determine the cause of this background. One possible source is substructure (dislocations and low angle grain boundary) introduced into the bicrystals by welding and handling. Figure 3 shows X-ray topographs of i) a single crystal of Nb as grown showing very little substructure, ii) one half of a bicrystal after welding but before annealing, iii) one half of a bicrystal after an anneal at 2100°C and iv) a bicrystal that has been handled in the process of attacking leads for the superconducting measurements. Welding primarily introduces very low angle subboundaries which can be reduced in number by the high temperature anneal. It would appear however that handling introduces at least as much additional damage. We are attempting to reduce these problems currently.

Flux pinning measurements have also been made on some of the asymmetric bicrystals in Table II. (These have a crystal anisotropy contribution of $\tilde{f}_p$). These have high critical currents at fields below .9 H_{c2}, too high to be measured with our current supply. In these bicrystals the results at high reduced field are complicated by the fact that the anisotropy of H_{c2} produces anisotropic pinning by the substructure, producing many peaks in addition to the grain boundary peak.

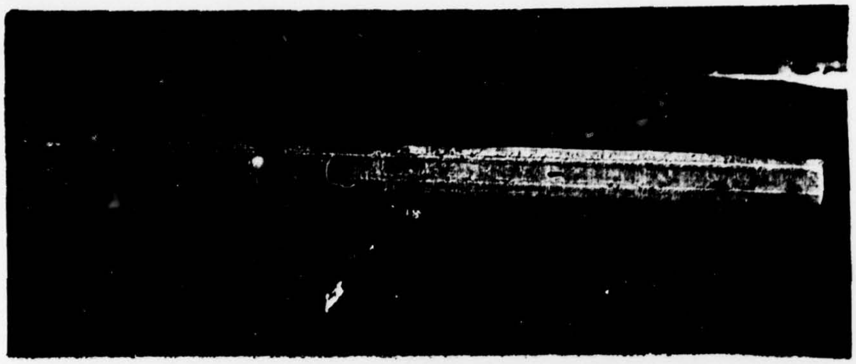
Transmission electron microscopic (TEM) observations of the structure of the grain boundaries has begun. A new thinning method (really a modification of an older method) has been developed by Dr. Schindler which avoids contamination of the section with hydrogen. The latter is important since we are beginning a collaboration with Dr. Uwe Essmann at the Max Planck Institute at Stuttgart where he will decorate the FLL in thinned foils of Nb containing the boundary for subsequent TEM observation and we would like the impurity content of such foils to be the same as the bulk bicrystal. Electron diffraction, weak beam methods and lattice imaging are being used to investigate the periodic (or non-periodic) nature of the boundary. Figure 4 shows a lattice fringe image of the grain boundary in bicrystal B. The boundary is inclined $\sim$15° from the normal to the foil. The terminating fringes are edge components of two dislocations which impinge on the boundary. No periodic structure could be detected in this high angle boundary. Conventional, lower resolution, TEM revealed that this grain boundary is straight without any facets or visible segregation of impurity species.

Figure 2. Critical current versus angle for a symmetric tilt bicrystal (bicrystal A).

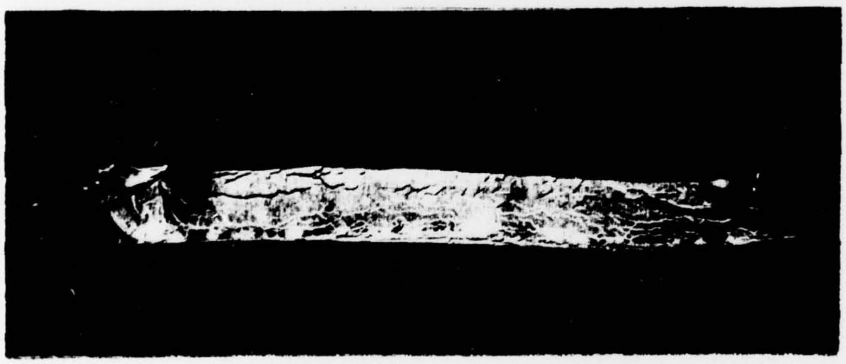


BERG-BARRETT X-RAY TOPOGRAPHY OF NIOBIUM BICRYSTALS

Figure 3. Berg-Barrett X-ray topographs of niobium bicrystals.

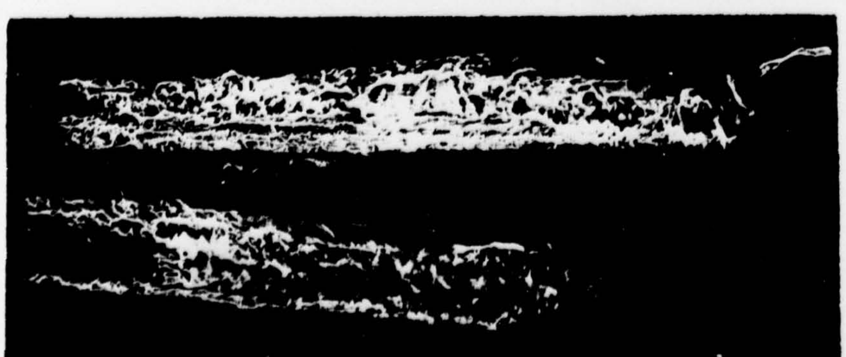


(i.) single crystal as grown



(ii.) bicrystal after welding ; no anneal

(iii.) bicrystal after 2100° C anneal



(iv.) bicrystal after voltage and current leads are attached

Figure 4. Direct lattice image of grain boundary in Nb bicrystal 3 (45° symmetric tilt). Insert shows electron diffraction pattern and the two $01\bar{1}$ reflections used to form the image.

In agreement with the X-ray topographs occasional subboundaries, approximately parallel to the high angle boundary, can be observed. The misorientation across these subboundaries is usually less than 0.5°. These observations are important for interpretation of the grain boundary critical current peaks. The ones we observe have a much larger angular half width than those reported by Das Gupta et al.[1] Measurements of I_c at very small angular intervals reveal that the I_c grain boundary peaks sometimes contain jagged subsidiary maxima. It is tempting to attribute these subsidiary maxima to the subboundaries. If this is true it indicates that low angle boundaries are somewhat weaker pinning centers (but not much weaker) than high angle boundaries.

2. Thin Film Polycrystal Studies

Polycrystalline evaporated films of $Pb_{82}Bi_{18}$ alloy composition provide a simple high κ system whose flux pinning in transverse magnetic field (F_p(GB)) is dominated by the grain boundary contribution. Pinning measurements in this system average over many random boundary misorientations; the critical field anisotropy effect should be small here, since the crystallites in such films tend to orient their <111> axes perpendicular to the film plane, and thus along a transversely applied field.

Last year it was discovered that striking changes occur in transverse pinning when $T\ell$ is introduced into the grain boundary (and adjacent volume) by diffusion at room temperature. Investigation of this phenomenon has continued and has also been extended to the Pb-$Pb_{82}Bi_{18}$ diffusion couple.

To distinguish the effects of such coating, the pinning in uncoated films must be well characterized. By tilting the substrate during deposition, and observing an identical shift in the magnetic field orientation at which F_p(GB) peaks, it has been shown that the grain boundaries tend to be oriented in the direction of deposition, as earlier assumed. The annealing procedure needed before the coating is deposited has been established, so that changes in F_p(GB) due to grain growth do not become confused with those due to coating penetration. By examining uncoated films it has been shown that the thermal cycling undergone during a series of anneals does not affect F_p(GB). Characteristic shapes of F_p(GB) vs H, and characteristic behavior with temperature, have also been established for uncoated films. A method of sputter etching to reveal the grain boundaries at the surface of the films has been developed which replaces an earlier, less sensitive, chemical etching procedure; used with a standard carbon replica technique for the transmission electron microscope, this method allows a correlation between F_p(GB) and the average grain spacing (Figs. 5 and 6) to be established.

Early in the studies of $T\ell$ coated films it became clear there was a problem in reproducibility; some films were strongly affected by coating, some hardly at all. A number of changes in fabrication and measurement technique have been made which alleviated this problem. Compared with earlier films, present films are thinner and are more thoroughly, uniformly, and reproducibly heat sunk to their LN_2 cooled holder during deposition; substrates are now tilted to obtain perpendicular incidence of evaporant. Present films are also smoother, have a smaller and more stable grain structure (with regard to grain growth or recrystallization), and are more reproducible from film to film.

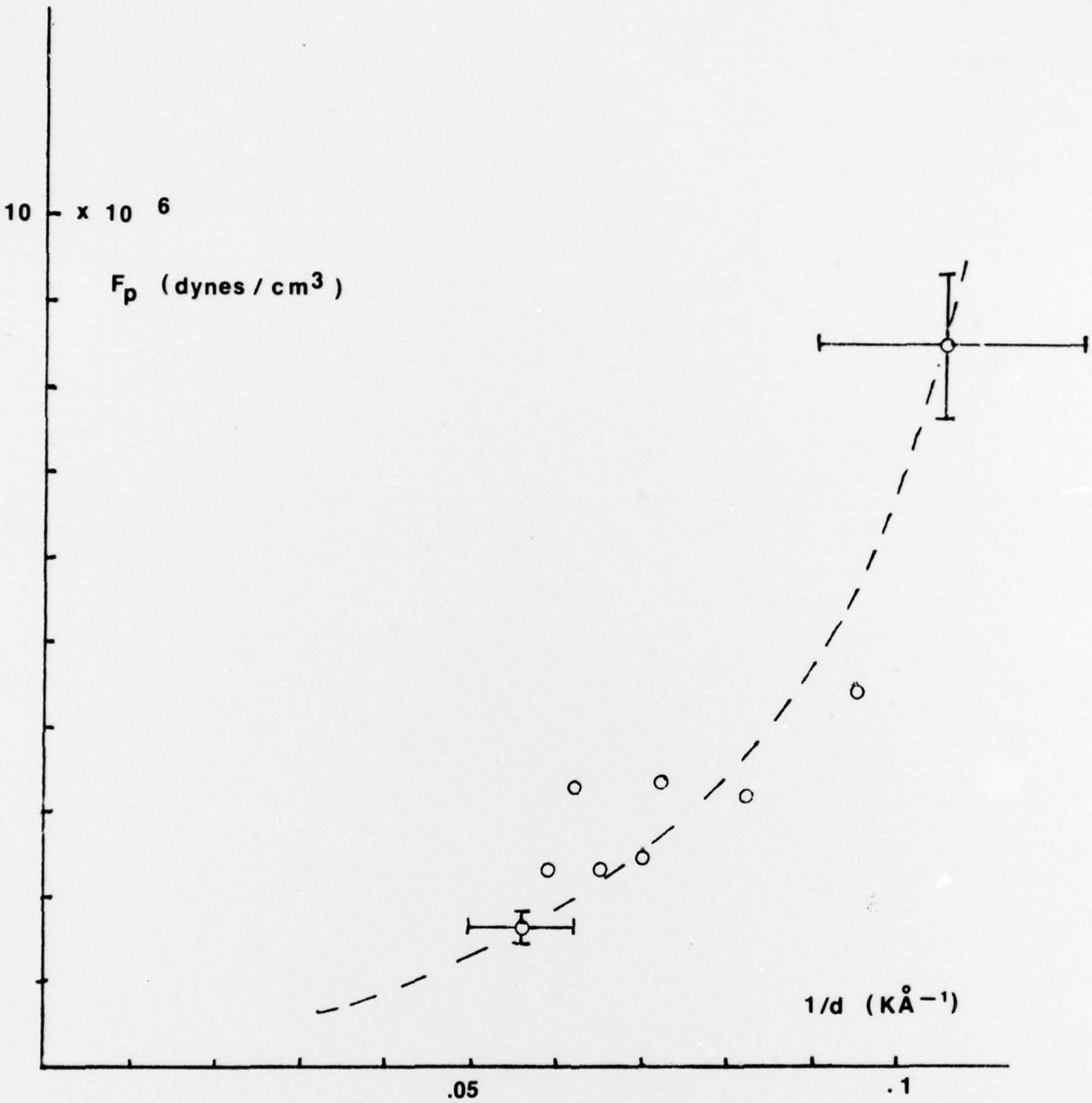


Figure 5. F_p(GB) at h = 1/3 for uncoated films vs 1/d, where d is the average grain boundary spacing.

Figure 6. Carbon replica of a typical sputter etched film; note the wide grain size distribution.

The largest problem, however, involves the formation of a diffusion barrier at the interface between coating and base. Ideally, one wishes to measure pinning on the same film before and after coating. Removal of the uncoated film from the vacuum of the fabrication system, however, results in sufficient oxide formation to block interdiffusion after coating. Even films left excessively long in the vacuum system seem to develop enough surface contamination to hinder diffusion. For films with rough surfaces, there is evidence that cracks in the interface oxide allow diffusion "leakage", but this process is highly erratic. Attempts were made to protect the film in an argon atmosphere during transfer to the testing probe and to apply the coating, after initial measurement, in an above-dewar vacuum chamber but these were not successful.

This problem has been circumvented by producing two films simultaneously and coating only one of the pair. Twin uncoated films have been found to agree closely in thickness, H_{c2} and $F_p(GB)$. The films are kept cold during removal from vacuum, scribing, and insertion into the resistivity test probe. A small amount of interdiffusion is seen in the initial measurement as a result of heating during the coating process.

Having eliminated these problems, some difficulties in the Tℓ coated system remain. Figure 7 shows the changes in pinning for 2 such films of different coating thicknesses. The complexity of the changes occurring -- (1) rapid and simultaneous changes in H_{c2}, H_c, and κ with composition, (2) coating oxidation, and (3) possible second phase formation -- make this system difficult to analyze.

The Pb-coated film system, however, has only two components (i.e., Pb and Bi) and there is no possibility of 2nd phase formation. H_{c2} is a sensitive but well known function of composition, while H_c is rather insensitive to composition. Oxidation does not seem to be a problem. Very thin Pb coatings produce dramatic, yet reproducible, changes in $F_p(GB)$ while changing H_{c2} little -- changes which are similar to those arising with Tℓ and probably produced by the same mechanisms.

Interdiffusion was carried out at temperatures close to 20°C (in this regime grain boundary diffusion is dominant). Penetration occurs very rapidly along the grain boundaries of the base film, then more slowly into the bulk of the grains, creating a network of Pb-rich zones which widen with time. From Figure 8 one sees that apparently the optimum pinning width varies with reduced field, peaking sharply for $h(\equiv \frac{H}{H_{c2}}) \leq .5$ then declining to near the uncoated twin value as homogenization is approached. In contrast, at high h the initial reaction is a strong dip in $F_p(GB)$. This high field reversal may result from formation of a lower κ path along the grain boundaries parallel to the direction of motion of the FLL where easy FLL shear can occur. If one examines the shift in time scale of these features with annealling temperature (see Fig. 9) one finds an apparent activation energy of roughly 60 kcal/mole -- much higher than expected for bulk diffusion (Pb-Pb diffusion has a Q of ~26 kcal/mole,[2]. Also, if the D = $2.5 \times 10^{-17} cm^2/sec$ for Pb-Bi interdiffusion[3] at room temperature is used, the resultant diffusion length at times corresponding to homogenization is much smaller than the minimum grain size observed. This result implies that some other mechanism may also be operating to broaden the composition profile at the grain boundary. One possibility is coated grain boundary migration driven by the free energy of mixing of the Pb in the Pb-Bi alloy.[4]

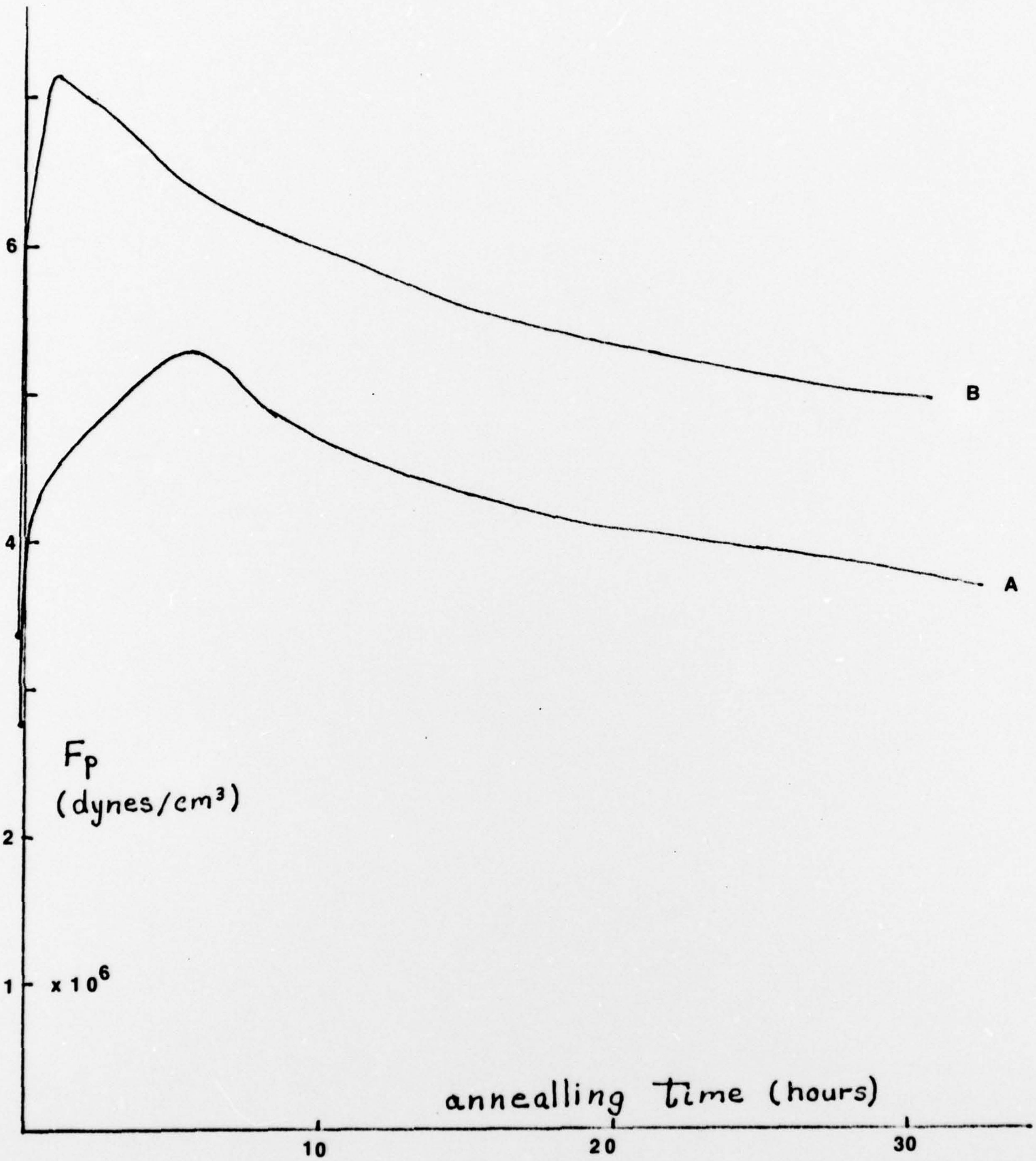


Figure 7. Thallium coated films: F_p(GB) vs annealling time at 18°C and h = 1/3; A - 150 Å coating, B - 1.8 kÅ coating.

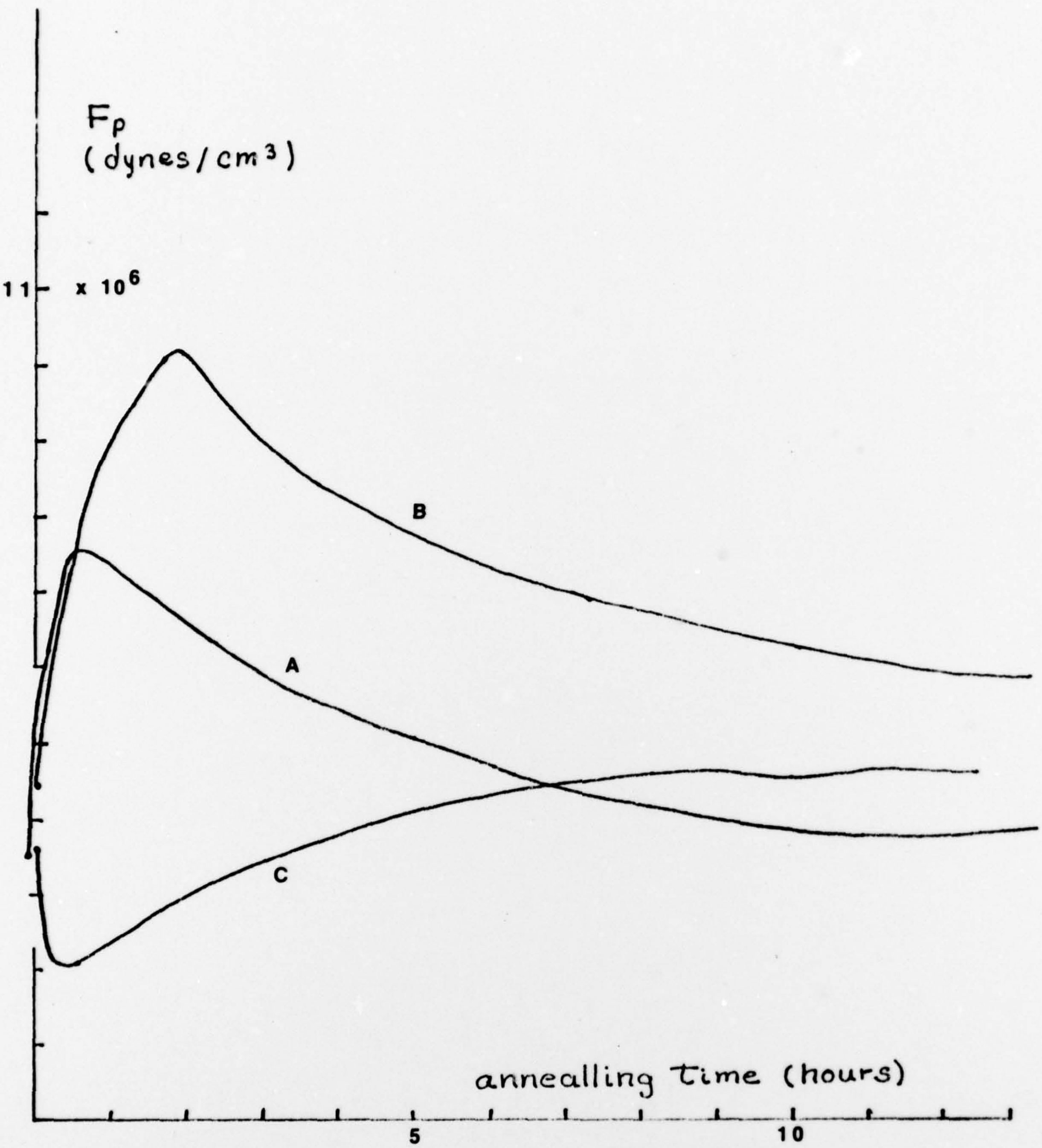


Figure 8. Typical lead coated film; F_p(GB) vs annealling time at 20°C; A ; h = .167, B ; h = .33, C ; h = .83.

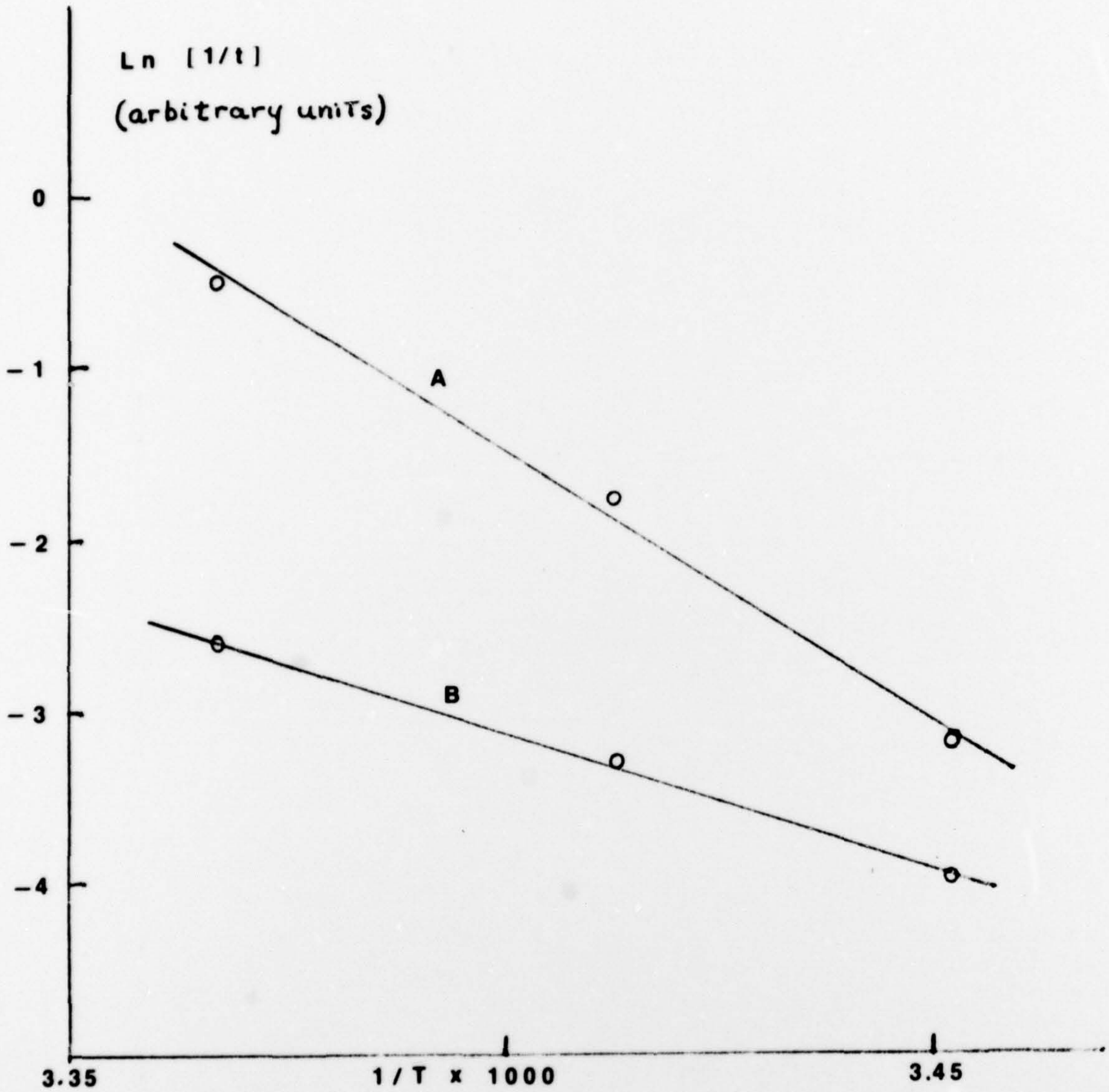


Figure 9. Ln of the rate of change of pinning force (expressed as [time to peak, t]$^{-1}$) vs inverse temperature for: A - grain boundary pinning F_p(GB) at h = 1/3; b - surface pinning $F_p(||)$ at h = 1/2.

Changes also occur in surface pinning. In contrast to the findings of Evetts[5] with the $T\ell$- $Pb/T\ell$ diffusion couple, the presence of normal metal on the film surface reduces $F_p(||)$ strongly (compared to the uncoated twin). However, just as Evetts observed, $F_p(||)$ first peaks and then declines as diffusion proceeds. The activation energy for both this process and final homogenization is $\sim$35 kcal/mole.

In both coated and uncoated films the shape of $F_p(GB)$ vs H remains roughly constant with temperature. When examined in detail however $F_p(GB)$ does not scale exactly as $F_p(GB) \propto H_{c2}{}^n(T)$ with constant n at all measured fields. Also n differs for different aging times and fields.

More work needs to be done to verify the above activation energies, to examine the effects of coating thickness and alloy composition of the base film. Attempts will be made to determine the microstructural changes occurring during interdiffusion by examining thin coated films in the transmission electron microscope. Ultimately it would appear that this system will be an excellent model for investigating the effects of $\Delta\kappa$ pinning due to grain boundary segregation on critical current densities. At the very least these experiments demonstrate unequivocally that grain boundary segregation in commercial high field superconductors must be seriously considered as a possible pinning mechanism.

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5) J.E. Evetts, Phys. Rev. B, 2 (1970) 95.

PERSONNEL

Principal Investigator:

Edward J. Kramer — B.Ch.E. with distinction, Cornell 1962; Ph.D. (Metallurgy and Materials) Carnegie-Mellon, 1967; NATO Postdoctoral Fellow, University of Oxford, Department of Metallurgy, 1966-67; Assistant Professor, Cornell University, 1967-72; Associate Professor, Cornell University, 1972 to 1979; Professor 1979 to present; Visiting Scientist, Argonne National Laboratory, 1974-1975.

Professor Kramer's research at Cornell has centered on the relationships between superconducting properties and metallurgical microstructure and on the mechanical properties and structure of polymers. He is author or co-author of over 45 publications.

In superconductivity his research has been primarily concerned with flux pinning in type II superconductors. His more than 20 publications in this area include experiments and theory on flux pinning by dislocations, surfaces, radiation damage (dislocation loops, voids, cascades and Frenkel defects), and grain boundaries. He has also published major papers on the summation problem, the problem of correctly summing elementary interaction forces to determine the global pinning force density. As a result of this activity he was asked to be a keynote speaker at both the International Discussion Meeting on Flux Pinning held in St. Andeasburg, West Germany, 1974 and at the International Discussion Meeting on Radiation Effects in Superconductors, Argonne, Illinois in 1977, as well as being an invited speaker on flux pinning at national meetings of the Metallurgical Society of AIME and the Materials Research Society.

Postdoctoral Associates:

Dr. Roland Schindler, Ph.D. Max Planck Institute für Metallforschung, Stuttgart, West Germany. Dr. Schindler worked with Professor Seeger at Stuttart on electron microscopy of radiation damaged metals. He has been a postdoctoral associate at Cornell, with Professor R.W. Balluffi, where he used electron microscopy to investigate the structure of grain boundaries in welded gold bicrystals. Dr. Schindler joined the research project in August 1978 and will stay one year until September 30, 1979.

Dr. Michael Lunnon, Ph.D. in Physics, University of Bristol, England. Dr. Lunnon worked with Dr. David Dingley using electron channeling methods to study the early stages of recrystallization and grain growth in copper. He brings substantial esperience in TEM and grain boundary structure to the project.

Research Assistants:

Mr. Wilson Yetter: Mr. Yetter is a graduate student in his fifth year at Cornell. He has been responsible for the manufacture of the Pb-Bi films as well as the measurements of flux pinning in these films. We expect that he will finish his Ph.D. thesis by September 1979.

Mr. Donald Thomas: Mr Thomas is a graduate student in his second year at Cornell. He has been assisting in the manufacture of Nb bicrystals as well as carrying out the preliminary flux pinning measurements on these bicrystals.

PUBLICATIONS UNDER THIS GRANT

E.J. Kramer, "Fundamental Defect-Fluxoid Interactions in Irradiated Superconductors," J. Nuclear Materials

E.J. Kramer, "Summation Curves for Flux Pinning in Superconductors," J. Appl. Phys. 49, 742 (1978)

B. Addis, D. Thomas and E.J. Kramer, "An Electron Beam Float Zone Welding Method for Preparing Refractory Metal Bicrystals," in preparation

RECENT INVITED TALKS AND COLLOQUIA ON FLUX PINNING

E.J. Kramer, "Flux Pinning in Superconductors - a New Approach to Summation," Harvard University, Solid State Physics Colloquium, October 7, 1977

E.J. Kramer, "Flux Pinning in Superconductors," Iowa State University, General Physics Colloquium, January 30, 1978

E.J. Kramer, "The Summation Problem Revisited," Iowa State University, Solid State Physics Colloquium, January 31, 1978

INTERACTIONS IN THE LAST YEAR

Dr. F. Habbal, University of Cincinnati, now at Harvard University

Dr. J. Thompson, Los Alamos Scientific Laboratory

Prof. Roger Rollins, Ohio University

Prof. Robert Reed, Penn State University

Dr. Amit Das Gupta, Oak Ridge National Laboratory
(Das Gupta is also working on flux pinning in Nb bicrystals and we keep in close touch so we do not duplicate efforts.)

Dr. S. Alterovitz and Dr. J. Woolam, NASA-Lewis Research Center

Dr. T. Francavilla, Naval Research Laboratory

Dr. Helmut Brandt	Inst. fur Metallphysik, Stuttgart, on leave at Iowa State Univ.
Dr. John Clem	Iowa State University
Dr. J. Bevk Prof. D. Turnbull	Harvard University
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Dr. H. Kerchner, Solid State Div., Oak Ridge National Laboratory

Dr. Si Foner, National Magnet Lab.

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